# ADVANCED MATERIALS AND CHEMICALS

Ford Motor Company Scientific Research Laboratory and General Electric Research and Development (Joint Venture)

# Thermoplastic Automobile Components Could be Easily and Affordably Recycled

Cyclic thermoplastics technology offered an attractive method for developing recyclable structural components for more fuel-efficient automobiles. The technology was unproven and technically risky, however, so private capital was not available for the research. Therefore, Ford Motor Company, General Electric, and five other partners proposed the project to the Advanced Technology Program (ATP), and, in 1992, the joint venture was awarded cost-shared funding for a four-year project. During the ATP-funded project, the joint venture pioneered the use of cyclic thermoplastics in automotive components and other parts. The liquid composite molding process developed during this project is now being used by Ford to manufacture parts for its Austin Martin model. Intellectual property resulting from the cyclic composition research was sold to Cyclics Corporation, which is using the process to produce products ranging from bicycle frames to infrastructure parts such as utility poles and offshore oil rigs.

#### **COMPOSITE PERFORMANCE SCORE**

(based on a four star rating)

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Research and data for Status Report 91-01-0178 were collected during October - December 2001.

# **Conservation Concerns Drive New Materials Development**

During the late 1980s and early 1990s, there was increasing interest in using new materials to achieve weight reductions in automobile manufacturing. This interest grew out of the need to conserve energy and recycle automotive materials. Polymer matrix composites, it was thought, could help the automotive industry address these issues.

There was increasing interest in using new materials to achieve weight reductions in automobile manufacturing.

During this period, the Ford Motor Company Scientific Research Laboratory had been working to develop and evaluate processes to produce large composite structures using polymer-based thermosetting resins systems. Meanwhile, General Electric Central Research (GE) was in the process of identifying properties in

molecular compositions of matter that would be utilized in resin development of thermoplastics. Given the research under way by both Ford and GE, tremendous potential existed for developing materials and processes to manufacture composite automobile structural parts through the use of cyclic thermoplastic technology.

In 1991, the world market for advanced polymer matrix composites was \$4.7 billion, with the U.S. share reaching \$2.6 billion or 54 percent of the world market. At the time, polymer matrix composites were predominantly used in the manufacture of military aircraft and missiles, although research indicated that these materials could be used in the automotive industry. Preliminary studies at Ford suggested that the use of carbon fiber/polymer composites could achieve weight reductions in steel body structures of between 34 percent and 65 percent, which could increase the annual market for composite materials by an estimated \$20 billion. Other potential applications included commercial aircraft, airframes and engine structures, non-defense space structures, sports and recreation

equipment, mass transit systems, offshore oil exploration and production, and industrial facility applications.

# Joint Venture Identifies Material that Meets World-Market Need

In May 2000, a committee of diplomats from the 15 European Union member countries and members of the European Parliament adopted the European Union Endof-Life Vehicle Law. This law requires that manufacturers pay all, or a significant part, of the costs of taking back and recycling the new cars that they sell. As early as 1994, however, Ford and GE had already identified thermoplastic liquid composites as a class of materials that could be easily and affordably recycled.

# **Existing Methods Lack Required Flexibility**

In 1992, when the joint venture submitted its proposal to ATP, thermoset polymer-matrix composites were prevalent in automotive component manufacturing. These components were fabricated using a process called liquid composite molding, which uses a preformed shell made from glass fibers combined with a molded foam core to create a pre-form in the shape of the final part. The pre-form is then placed in a heated steel mold and clamped in a low-pressure press. Liquid resin is delivered from a pressurized pump into the mold and, once the resin is cured, the part is removed.

Traditional thermoplastics have been excluded from the liquid composite molding process because they are relatively viscous when melted. This viscosity creates difficulties in establishing a smooth flow of the material through the mold.

Thermoset polymers, when subjected to heat, undergo an irreversible chemical reaction, causing the molecules in the polymer to cross-link, or connect the polymer chains. As a result, the entire finished part becomes a single, large molecule. Thermosets are used in liquid composite molding, in large part because of processing issues. The low viscosity of unreacted thermoset resins facilitates rapid impregnation and wet-out (elimination of voids or imperfection of bonding) of the pre-form. Large, thermoset-based parts are difficult to manufacture, however, because of the heat (exothermic) generated in the process. This heat can reduce the finished product's

ability to withstand damage (i.e., its crashworthiness). In addition, thermoset-based parts are difficult to recycle.

Thermoplastics, in contrast to thermosets, can be recycled simply by reheating the material. Traditional thermoplastics have been excluded from the liquid composite molding process, however, because they are relatively viscous when melted. This viscosity creates difficulties in establishing a smooth flow of the material through the mold, even if high pressure is used.

# **Cyclic Technology Offers Improvements**

Like thermosets, cyclic thermoplastics exhibit low viscosity when molten so that they flow like water and low-injection pressure can be used. Moreover, unlike thermosets, cyclic thermoplastics do not have production problems associated with heat generation, which enhances their suitability for use in manufacturing large component parts. Cyclic thermoplastics also are recyclable.

# **Seven-Member Project Team Obtains ATP Funding**

In 1992, a seven-member joint venture submitted its proposal to ATP and received a \$5.3 million, four-year award. It proposed to focus its efforts on the use of cyclic thermoplastics in liquid composite molding, also known as resin transfer molding. The joint venture included the following members:

- o Ford Motor Company, for automotive research
- General Electric, a major materials supplier and the proprietary owner of the technology
- Pittsburgh Plate Glass, a supplier of glass fiber materials
- American Lisitritz, a composites molding equipment manufacturer
- Rensselaer Polytechnic Institute, for engineering studies
- o The University of Tulsa, for recycling research
- The Environmental Research Institute of Michigan, to study the transfer of cyclic thermoplastic technology to parts fabricators

#### **Pilot Production Proceeds**

The project team concentrated on laboratory studies of resin chemistry and research and acquisition of a sophisticated melting device. Additionally, it undertook the initial fabrication of a laboratory molding system. Extensive discussions then ensued among team members regarding initiator systems for use in the process, ultimately resulting in the decision to use a low-viscosity liquid as the initiator. Not only were these initiators readily available, but the use of a liquid initiator was preferable because it enabled mixing to occur as the initiator and resin entered the mold.

The next step was research selection and acquisition of an extruder that had the ability to melt and convey the polymer according to the team's specifications. At the conclusion of this step, and with the identification of a process and a primary material, the team began pilot production, which included the following:

- Developing and completing the laboratory molding system
- Continuous monitoring of the processing data generated by the system as development began
- Achieving production of cyclic resin in the required quantities
- Molding and testing of cyclic plaques
- Redesigning and developing a mixing head capable of metering the extreme ratios required

# Reevaluation Leads to Changes in Production Approach

The information and data generated throughout pilot production led to the installation of a large-scale system at Ford within the original timeline, as well as the actual production of a prototype. Once production was under way, the team reviewed the prototype results. Actual molded plaque data and cost projections revealed the need to reevaluate the cost approach to resin synthesis and the originally projected physical properties, including recycling potential, in molded composites.

Subsequently, the team carefully studied and then selected a potentially lower cost approach to resin synthesis. With the modifications and adjustments to the process in place, and the project now back on track, the team produced acceptable test plaques in sufficient quantity and quality. Preliminary testing, however, determined that the properties of the cyclic plaques did not meet the established baseline requirements of thermosets. At this juncture, researchers experimented with and evaluated a range of initiators, stabilizers, and antioxidants that could improve the properties of the cyclic thermoplastics.

During this evaluation period, the team selected a core technology and a supplier, and it continued molded component production. Evaluation of the product indicated that the team had achieved the best properties thus far through adjustments in resin metering and mixing. As the project drew to a close, the team manufactured six composite cross-members, three of which were testable.

# **ATP Support Accelerates Technology Development**

Ford and GE determined that the ATP funding accelerated the development of the resin system and processing technology by two to four years. Since private capital was not available because of the risks associated with the project, the joint venture would not have been able to set up and operate a project of this magnitude without ATP funding.

With ATP's encouragement, Ford and GE were able to recognized the importance of their research for the entire automotive community and shared performance data through the auspices of the Automobile Composites Consortium (composed of Ford, General Motors, and DaimlerChrysler), thus enhancing the entire industry's understanding of the liquid composite molding process.

### Conclusion

At project completion in 1996, the research team had met its manufacturing cost target of approximately \$1 per pound. The team did not, however, achieve its goal of translating key properties from laboratory beaker reactions to materials made under simulated production conditions. Additionally, the joint venture had not fully demonstrated key resin attributes, such as toughness and energy-absorbing capability. As a result of ATP funding, however, the project achieved success in the following areas:

- Substantial data were collected regarding mold flow and filling.
- Eighteen patents were awarded.
- Six cross-member structural parts were manufactured using the liquid composite molding process (although only three were testable).
- Ford is currently using the liquid composite molding process.

In 1999, privately owned Cyclics Corporation, headquartered in Rensselaer, New York, received funding and purchased GE's portfolio of patents related to cyclic thermoplastics. The company has continued to enhance the technology and has developed alliances to manufacture and market resins for applications in the automotive, construction, and powder-coating industries. Cyclics Corporation is also undertaking a number of development projects for direct customers in structural composites and related technology areas.

# **PROJECT HIGHLIGHTS**

# Ford Motor Company Scientific Research Laboratory and General Electric Research and Development (Joint Venture)

**Project Title:** Thermoplastic Automobile Components
Could Be Easily and Affordably Recycled (Cyclic Thermoplastic
Liquid Composite Molding for Automobile Structures)

**Project:** To develop a cost-effective use of cyclic thermoplastic composites as structural components to produce more fuel-efficient automobiles.

**Duration:** 7/15/1992-12/31/1996 **ATP Number:** 91-01-0178

# Funding (in thousands):

ATP Final Cost \$ 5,292 48%
Participant Final Cost 5,735 52%

Total \$11,027

Accomplishments: The research conducted by the joint venture led to the collection of substantial data regarding mold flow and filling. This information, which the joint venture shared with the Automobile Composites Consortium, enhanced the automobile industry's understanding of the liquid composite molding process. In addition, the research led to the award of 18 patents for the materials and manufacturing processes developed during the ATP project. Moreover, Ford currently uses the liquid composite molding process. The joint venture was unable, however, to manufacture cyclic thermoplastic cross-member structural component parts that tested above the baseline indicators of like-constructed thermosets. Although the joint venture manufactured six cross-members from cyclic thermoplastic material by using the liquid composite molding process, only three of the six were testable.

The following patents were awarded as a result of this ATP project

- "Macrocyclic polyester oligomer preparation with Lewis acid as catalyst"
   (No. 5,321,117: filed November 19, 1992, granted June 14, 1994)
- "Process for making thermoplastic polyester foams"
   (No. 5,348,985: filed January 18, 1994, granted
   September 20, 1994)
- "Trisstannoxanes useful for polymerizing macrocyclic poly(alkylene dicarboxylate) oligomers"
   (No. 5,386,037: filed June 20, 1994, granted January 31, 1995)

- "Branched polyesters prepared from macrocyclic poly(alkylene dicarboxylate) oligomers" (No. 5,387,666: filed June 20, 1994, granted February 7, 1995)
- "Method for polymerizing macrocyclic poly(alkylene dicarboxylate) oligomers"
   (No. 5,389,719: filed June 20, 1994, granted February 14, 1995)
- "Resin molding process utilizing a core prepared from glass beads and a binder"
   (No. 5,492,660: filed August 1, 1994, granted February 20, 1996)
- "Depolymerization process using tin catalysis. Not titanates included" (No. 5,407,984: filed August 31, 1994, granted April 18, 1995)
- "Process for preparing macrocyclic polyesters"
   (No. 5,446,122: filed November 30, 1994, granted August 29, 1995)
- "Process for removing linears from cyclics with sieves"
   (No. 5,434,244: filed December 9, 1994, granted July 18, 1995)
- "Polymerization of macrocyclic poly(alkylene dicarboxylate) oligomers"
   (No. 5,466,744: filed January 9, 1995, granted November 14, 1995)
- "Method for polymerizing macrocyclic poly(alkylene dicarboxylate) oligomers"
   (No. 5,527,976: filed January 12, 1995, granted June 18, 1996)
- "Method for polymerizing macrocyclic polyester oligomers" (No. 5,498,651: filed June 19, 1995, granted March 12, 1996)
- "Method for polymerizing macrocyclic poly(alkylene dicarboxylate) oligomers"
   (No. 5,591,800: filed November 30, 1995, granted November 30, 1997)
- "Process for producing high ductile polyesters"
   (No. 5,648,454: filed February 14, 1996, granted July 15, 1997)

- "Process for depolymerizing polyesters"
   (No. 5,668,186: filed March 20, 1996, granted September 16, 1997)
- "Titanate catalysts"
   (No. 5,710,086: filed March 20, 1996, granted January 20, 1998)
- "Titanate esters useful as polymerization initiators for macrocylic polyester oligomers" (No. 5,661,214: filed August 5, 1996, granted August 26, 1997)
- "Method for producing polyesters"
   (No. 5,663,282: filed August 19,1996, granted September 2, 1997)

Commercialization Status: Ford is currently using the liquid composite molding process to manufacture 30 component parts for its Aston Martin model. In June 1999, Cyclics Corporation, of Rensselaer, New York, bought 50 U.S. and foreign patents from GE. These patents cover cyclic compositions, technology that GE had developed with partial funding from ATP. Cyclics Corporation will market its Cyclic Resin Systems technology and is currently using the technology to produce recyclable bicycle frames, utility poles, and other infrastructural parts.

**Outlook:** The research conducted on the technology and the processes yielded valuable information to help bring cyclic thermoplastic technology to market. As a result of this ATP project, the manufacture of cyclic thermoplastic automobile parts is under way. Depending on regulatory developments regarding fuel economy and environmental concerns, further development of this technology could take place in the future.

Currently, Cyclics Corporation uses the technology to manufacture environmentally friendly products that range from bicycle frames to infrastructure parts such as utility poles, supports for bridges, and offshore oil rigs.

Composite Performance Score: \* \* \*

### Companies:

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#### **Subcontractors:**

- Pittsburgh Plate Glass
- American Lisitritz
- o Rensselaer Polytechnic Institute
- The University of Tulsa
- The Environmental Research Institute of Michigan